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LA-UR--87-2449

DE87 013161

TITLE: Using Rapid Solidification Processing to Produce Microstructures with Optimum Properties

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SUBMITTED TO: To be published in Proceedings of "Rapid Solidification Processing Principles and Technologies IV"

Conf. held Dec. 1986

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USING RAPID SOLIDIFICATION PROCESSING TO PRODUCE MICROSTRUCTURES WITH OPTIMUM PROPERTIES

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ABSTRACT

Rapid Solidification Processing (RSP) permits an additional degree of freedom for tailoring the microstructure of materials. As a result microstructures can be controlled to a degree which has not been consistently achieved by ingot metallurgy methods. This capability provides an opportunity to "forward engineer" the microstructure of materials so as to optimize a particular property or set of properties. When the choices of microstructure are required to made up front, it turns out that the bases for making them are often not well-founded in fundamentals. Thus, exploitation of RSP is promising but presently incomplete.

In this paper we review the principles for selecting microstructures and attempt to identify those areas where our understanding is incomplete. We also discuss some cases where these microstructural choices can be made and some where they cannot. Further, we describe some specific examples of alloy systems where RSP has been used successfully to tailor properties. Finally, we will attempt to assess the potential for extending these techniques to other alloy systems and to define some of the current limitations which are associated with RSP. This discussion will permit identification of those areas where additional research on processing-microstructure and microstructure-property relations is indicated.

INTRODUCTION

The rapid heat extraction associated with rapid solidification* processing (RSP) allows the production of assolidified microstructures with a size scale which can be several orders of magnitude finer than those that can be obtained through conventional ingot metallurgy methods. Along with this beriefit comes rather severe natural restrictions in product form, viz. the molten liquid (and thus the as-solidified product) must have at least one small dimension to permit an adequate heat extraction rate. Thus "as-RSP'd" materials typically exist as thin sheet or particulate, representing one and three small dimensions respectively. Wire (representing two small dimensions) also is possible but is technologically difficult to produce because of the inherent instability of small diameter liquid streams.

In addition to processes where solidification occurs throughout the entire volume of the material at once (bulk RSP), there are other processes which permit the achievement of high local solidification rates by sequential melting or deposition of thin layers which can be built up into bulk shapes (sequential RSP). Sequential RSP methods offer attractive alternatives to bulk methods because subsequent consolidation step is not required after solidification is complete. Thus there is less concern about coarsening and other unwanted microstructural changes which can impair realization of the benefits of RSP in final consolidated products.

Notwithstanding these potential benefits, the majority of effort expended to date in exploiting RSP for structural applications has been concentrated on the bulk processes. While, from a microstructural standpoint, bulk and sequential methods don't appear to be qualitatively different, they are clearly different from a post-solidification processing standpoint. Such considerations will ultimately determine which of the possible processes are acceptable for a particular application. Thus, while the close ties between overall processing and microstructure must be kept in mind in any discussion of RSP, detailed consideration of this point lies outside the intended scope of this paper.

There are also alternative processes not related to RSP per se which allow the synthesis of novel microstructures. Among these are vapor deposition and amorphization by mechanical mixing, either on a macroscopic scale by mechanical alloying or on a microscopic scale by irradiation. The best known vapor deposition method is that used by Gleiter (1) to make "nanocrystals", i.e. materials with grain sizes on the order of tens of nanometers. These materials have been shown to have interesting physical properties while only limited mechanical properties have been reported. Unfortunately, these materials have only been produced in very small quantities so they must be regarded at present as somewhat exotic and experimental in nature. As a result only limited comparisons exist between these materials and those produced by either conventional (ingot) or RSP methods.

In this paper we intend to discuss the production of selected microstructures by RSF and compare the capability that RSP provides for producing these structures to that of other, more conventional, methods such as ingot metallurgy. We also will suggest microstructural features which are desirable for optimizing particular properties and attempt to provide a rationale for why these features are desirable. The focus of our discussion will be monolithic materials, although the use of RSP in producing matrix materials for incorporation into metal matrix composites also is a promising area which has received less attention. We will, in addition, attempt to identify those areas where real benefits of RSP have already been demonstrated and to distinguish these from other areas which have been widely discussed but where no real benefit has (as yet) been realized. We also will attempt to identify a few unresolved issues which are limiting the acceptance and growth in application of RSP technology.

^{*} in this paper we will use rapid solidification to connote solidification at large undercooling. Under these conditions the redistribution of solute during freezing can be minimal.

ELEMENTS of MICROSTRUCTURE

If one accepts the concept that a hierarchy of structure exists which starts at the atomistic scale, several levels of description of microstructure can be accomplished. The following paragraphs will begin by describing microstructure at the finest scale and will end with a discussion of the strengthening potential of each of these distinct elements.

At the level of the atom, compositional variations (segregation) effects dominate the microstructure. If the solute atoms are perfectly uniform in their distribution, they are referred to as solid solution "strengtheners". Anything besides a random solute distribution implies some level of clustering or short range ordering. When this occurs on the size scale of several nanometers, it is not usually considered detrimental. However, during solidification, thermodynamic driving forces cause solute redistribution between the solid and the liquid on a size scale which depends on the cooling rate, but which is large compared to the atomic scale. Cellular breakdown can cause submicron rearrangements in composition, while dendritic structures produce micron scale inhomogeneities. As the cooling rate decreases, more macroscopic chemical segregation can occur as is often observed in conventional casting.

When the chemical inhomogeneities reach critical size and concentration levels, second phases are precipitated within the original structure. Classification of these precipitates can be accomplished by studying their method of formation. If they are pure elements, ceramics or intermetallic compounds and form directly from the melt due to minimal solubility in the solid matrix at any temperature, they are usually called dispersoids. Once dispersoids are formed, their distribution cannot be significantly altered by thermal treatment because of the minimal solubility of the components in the matrix phase (implying resistance to size and shape coarsening). If the second phase to form is soluble at some intermediate temperature, they are usually referred to as precipitates in the normal context of precipitation strengthening systems. The size and morphology of these second phases are determined by such fundamental factors as the relative surface energies of the two structures, their crystallographic mismatch, the chemical supersaturation and the diffusivities of the constituent atoms. These factors affect the nucleation and growth of the second phase precipitates and are strongly influenced by the thermal history of the material. Thus, in rapid cooling, the matrix may retain the metastable single phase until deliberate treatments are subsequently employed to precipitate the second phase. Two potential benefits of rapid cooling are: i) increased supersaturation (undercooling) of the precipitating species leading to a wider range of volume fraction and size of second phase particles during aging or; ii) the achievement of such large supersaturations that unanticipated metastable phases can be formed. Different alloy systems exhibit varying propensities for extension of equilibrium phase diagram solvii or new metastable phase substitution. This is currently an expanding area of study as has been described by Boettinger and Perepezko (2) elsewhere in this conference proceedings.

In addition to solutes, line and point defects will have a major effect on the subsequent properties. The concentration and arrangement of these defects are the result of processing history including mechanically and thermally induced strains. In the neighborhood of second phase precipitates or dispersoid particles, dislocations can be nucleated due to differences in the elastic-plastic behavior or thermal expansion coefficients between the matrix and second phase particles. Further, thermomechanical treatments (TMT) can result in controlled substructure near second phases in addition to the matrix dislocation substructure that would be present if it were a single phase material.

The grain size and shape also reflect processing history, including solidification rate, and can have a significant effect on subsequent properties. In the case of stable (noncoarsening) dispersoid containing RSP materials, the final grain size is strongly influenced by both the densification technique / parameters and the solidification rate of the particulate. In other RSP materials strengthened by solid solution or precipitation, the grain size after consolidation largely depends on the consolidation process and parameters. In this regard,

there is no persuasive evidence in the literature to support the contention that solidification history significantly affects solid state microstructure after a typical consolidation schedule. However, if the goal is to achieve fine grain sizes from particulate precursors, then the smallest particles, the lowest feasible densification temperature and the highest rate compaction technique would be the most likely means of achieving this end. A limiting example would be ultrafine powder and dynamic compaction. Grain shape also is important but mainly depends on mechanical deformation. Grain shapes other than equiaxed can impart anisotropy to the material. For example, thin plate shaped grains may not require five independent slip systems to accommodate specific strain paths (3).

A separate issue sometimes relaied to grain shape, but one which is often identified with it, is the distribution of crystallographic orientations (texture) in an aggregate of grains. Texture strongly affects a whole range of mechanical properties including yield stress, rate of work hardening and the propensity for plastic instability (4). Preferred orientation can arise from working, from recrystallization following working or from solidification. As the elastic and plastic anisotropy of the single crystal increases, the effect of preferred crystallographic orientation on macroscopic properties becomes more pronounced.

MICROSTRUCTURAL EFFECTS on SELECTED PROPERTIES

From the above it is clear that there are a number or microstructural features which have the greatest effect on properties of metals and alloys, but these can really be grouped into several categories as follows:

- · Solute Atoms substitutional and interstitial;
- Particles precipitates and dispersoids;
- · Boundaries grain boundaries and sub-boundaries;
- Texture type and intensity:
- Grain Size and Aspect Ratio.

It is beyond the scope of this paper to analyze the detailed effects of each of these microstructural elements on the general phenomena of mechanical behavior. The reader is referred to earlier reviews (5,7) for this discussion. An important grouping of some of these microstructural elements has been suggested by Argon (6). He divides hardening mechanisms into diffuse and primary. Among the various hardening mechanisms used to improve the strength of metals, the only one which clearly qualifies as a primary mechanism is strengthening by dispersoids. On the other hand, texture (4), solid solution, grain size and precipitation hardening all are diffuse hardening mechanisms. Primary hardening mechanisms are important because they lead to strength improvements without increasing the tendency for plastic instability. Thus primary hardening by dispersoids is attractive since it has the potential for increasing both the yield strength and the work hardening rate, the eby inhibiting shear localization and leaving the toughness unaffected.

However, it is clear that RSP can be used to alter only some of the above mentioned microstructural features. This is in part because of the scale on which these features occur and in part because of the intrinsic origins of the different microstructural features. For example texture and grain aspect ratio are usually produced by thermomechanical processing and are more readily altered by TMP than by RSP. The remaining features can be affected to a varying extent by RSP. In the following section we will discuss the effect of RSP on properties of selected alloys by considering the effect that this processing method has on their microstructure.

Grain Size Effects

One of the principal benefits of using RSP is the nearly intrinsic refinement of grain size. Thus it is useful to consider the effect which smaller grains have on properties. We will consider two classes of properties; those which have been traditionally studied under monotonic loading in connection with grain size variations and those which are related to cyclic loading. We will term the former static properties and the latter cyclic properties.

Static Properties: Because RSP permits the production of materials with small (≈1μm diameter) grain sizes, there is a basis for renewed interest in the effects of grain size on properties at smaller grain sizes (<1μm) than have been examined in detail over the years. Several questions seem important in this context.

- First, does the Hall Petch (YS \approx d-1/2) relation hold at smaller (sub-μm) grain sizes?
- Is there a lower limit or cut-off below which this relation does not hold?
- What happens to the work hardening rate at sub-micron grain sizes?
- How do the variations in strength and work hardening affect properties such as ductility and toughness?

There is conflicting evidence regarding the applicability of the Hall-Petch relation at small grain sizes. One of the reasons for this is that prior to the availability of RSP, there were no straight forward ways to produce materials with sub-micron grain sizes. As a result, electrodeposition and severe mechanical working followed by recrystallization were the most commonly used methods. Each of these methods introduces "side effects" in the form of impurities and second phases or preferred orientation (texture), both of which affect the strength. This makes it relatively difficult to determine the strengthening contributions due to grain size variations alone in materials where other factors vary simultaneously with the grain size. Notwithstanding these complications, work reported on electrodeposited Ni and Ni-Co alloys suggests that the strength of these alloys does not obey the Hall-Petch relation at grain diameters below ≈1µm(8). This situation differs from that for dislocation cells where the strength has been shown to follow a 1-1 relation down to cell sizes as small as 0.25 µm (9,10), where I is the cell size. The effect of "barrier strength" on the Hall-Petch exponent has been used to rationalize the differences between cells and subgrain boundaries(9,10,11). That the stronger barriers such as high angle boundaries lead to d-1/2 behavior is supported by data on layered composite materials consisting of Al and Al₂O₃ where the metallic slip length is as small as 50nm (11,12). It is wellknown that the work hardening rate decreases with small grain sizes. This tends to promote plastic instability and leads to reductions in tensile ductility and toughness. Part of the reason for this has been discussed by Thompson (11), who suggests that the progressive decrease in work hardening with strain in a conventional fine grained material (d values in the 5-10 µm range) is related to the development of a preferentially deformed zone near grain boundaries. This zone expands into the grain interior with increasing strain; the strain at which this zone engulf the entire grain volume depends on the initial grain size. As a result, the smaller the grain size, the lower the strain at which the work hardening saturates (9,10). Saturation in work hardening is related to the onset of plastic instability as predicted by the Considere' criterion. Processes which occur at a critical local strain, e.g. ductile fracture, may thus exhibit a different grain size dependence than those processes which depend more on overall plastic strain, e.g. tensile ductility (at least prior to the onset of necking). There is a need for additional experiments on very fine grained materials to examine the grain size dependence of toughness for grain sizes in the range of =10nm to 1µm. The ability of RSP to routinely produce materials with grain sizes in this range suggests that this is an opportunity for practitioners of RSP to pursue.

Alloys which fracture by cleavage rather than by microvoid nucleation, growth and coalescence can have a different dependence of toughness on grain size, especially if the range of grain sizes spans the dimension of the Griffith crack size. At this size, microcracks which are formed instantaneously become propagating cracks and lead to rapid macrocrack formation and fracture. In brittle metallic materials which fracture by cleavage

(examples include bcc metals below the ductile-brittle transition temperature, or intermetallic compounds such as Ti₃Al) are usually tougher at smaller grain sizes. However, this depends on the grain size dependence of the work hardening rate. For materials which fracture by cleavage, the work hardening dependence of the fracture resistance is generally opposite to that of ductile materials because the operative fracture criterion is one of critical local stress as opposed to critical strain. Thus high work hardening causes rapid elevation of the stress level. This reduces the critical crack size and promotes the onset of unstable brittle fracture. In contrast, a high work hardening rate in ductile metals stabilizes plastic flow and defers fracture to higher strains where the work hardening rate decreases due to exhaustion of the work hardening capacity, as mentioned earlier.

Cyclic Properties: Grain size also affects cyclic properties such as fatigue crack initiation (FCI) and fatigue crack growth (FCG). Thus in addition to the effects on strength and toughness mentioned above, RSP has the potential for tailoring these properties. The principal factors which affect FCI and FCG are the following:

- · Yield Strength;
- · Slip Character;
- · Slip Length;
- · Strain Localization;
- · Cyclic Hardening/Softening.

Among these, grain size directly affects yield strength and slip length directly whereas it only affects strain localization and cyclic hardening /softening in a second order way. Slip length has a large effect on fatigue crack initiation, as slip length decreases the number of cycles to crack initiation increases. This effect is particularly pronounced in materials which exhibit planar slip, e.g. alloys which contain coherent precipitates that are sheared during deformation or alloys which have short or long range range order in the matrix. In ordered alloys such as intermetallic compounds the role of grain size is very important in crack initiation with smaller grain sizes leading to improved fatigue lifetimes.

In FCG grain size also has an effect but it generally is opposite to that on FCI. That is, FCG generally is reduced at a given ΔK level. The reason for this appears to be related to an increased fracture roughness in larger grained materials, especially at lower crack growth rates, e.g. $\leq 10^{-8}$ m/cycle. Increased roughness leads to higher crack closure loads which in turn results in a lower fraction of the applied ΔK being available as a driving force to extend the crack. The extent to which grain size alters roughness also depends on slip character, in general, the more planar and localized the slip, the rougher the fracture surface.

The foregoing makes it clear that grain size can have a pronounced effect on the fatigue performance of RSP materials but it also is clear that the grain size effects are different for the two principal fatigue properties, FCI and FCG. Thus the choices for structure and thus the potential value of RSP depends on which fatigue property is the design pacing one.

Other Microstructure Effects

In addition to grain size, there are other microstructure features which are affected by RSP and which can influence the properties of the final product. Included would be small, uniformly distributed dispersoids which can be formed in alloys produced by RSP. These dispersoids can lead to direct increases in strength by acting as rigid barriers to dislocation motion. The strengthening contribution from these dispersoids comes from the Orowan mechanism and therefore is proportional to the shear modulus and inversely proportional to the

particle spacing. In Ti and Al alloys, which have lower moduli (by a factor of 2 or 3), than Fe, Ni or Co, the required spacing to achieve a comparable increment in strength is smaller by the same factor. This poses a relatively severe limitation on the maximum dispersion strengthening contribution which can be realized in the se alloys. This limitation is even more severe if particle coarsening occurs during consolidation or service.

Dispersoids also have two other effects: they act as grain refiners and they increase the work hardening rate. In the case of the former, the benefits to fracture related properties such as ductility and toughness have been discussed. For materials intended for elevated service the constraints on grain growth imposed by the dispersoids can lead to a significant penalty in creep performance, particularly in the temperature regime where grain boundary sliding or diffusional creep dominated the creep rate. The dispersoids also limit the extent to which special processing such as directional recrystallization can be successfully employer to eliminate transverse grain boundaries, in components such as turbine blades and vanes. With regard to the effect of dispersoids on work hardening rate, this is helpful in reducing the propensity for plastic instability in alloys which fracture by microvoid growth and coalescence. However, in alloys which fracture by a normal stress controlled mechanism such as cleavage, the increase in work hardening rate rapidly raises the flow stress which tends to favor the incidence of fracture. From this discussion it should be clear that dispersoids are not a panacea for improving the performance of RSP processed materials. Thus those individuals involved in selection of microstructures of advanced materials should think carefully about the intended use of the materials before choosing compositions and processing routes. Failure to do so can easily result in optimization of the wrong property or at least one which is of secondary importance.

ROLE of RSP in MATERIALS ENGINEERING

Given the impact of the various microstructural features on these selected properties, it is pertinent to discuss how RSP can affect the microstructure of alloys and thus their properties. Examples from several alloy systems will be used to emphasize how the advantages of RSP processing have been successfully utilized.

Rapid solidification can lead to a supersaturated solid solution thus extending the maximum solid solution strengthening available to a system. The metastable nature of this configuration means either that the RSP naterial must be produced in the final product form (ribbon or foil) or that densification is carried out at low emperatures / short times so that nucleation of the equilibrium product is avoided. Utilization of RSP to exceed the equilibrium solid solution strengthening is unusual due to the product form criteria (neglecting the amorphous magnetic applications). The more conventional application of increasing the solid solubility of solute is in the area of precipitation strengthening. By exceeding the supersaturation attainable by conventional casting, the volume fraction of second phase particles can be significantly increased. The listribution of these precipitates can be controlled with the classical time and temperature considerations of nucleation and growth if the densification step does not cause decomposition. A detailed analysis of the precipitation process in material which has undergone different cooling paths, such as melt spun ribbon zones \ and B or microcrystalline/cellular/dendritic powder structures, has not as yet been reported (14). lowever, it stands to reason that the degree of supersaturation should be a function of cooling rate and thus the recipitation reaction that follows should produce different volume fractions, if not different size listributions, in these various solidification structures. The goal of attaining a more uniform distribution of solute through rapid solidification has been attained if the comparison is made to conventional casting type nhomogeneities, but finer and finer scale segregation might effect precipitation reactions.

Significant effort has been expended in the effort to produce a uniform distribution of inert dispersoid particles in several different alloy systems (15,16,17,24). During solidification, the first solid to form in a plant alloy does so after the temperature decreases below the liquidus. When the alloy contains the necessary constituents (such as ternary elements) to form a refractory intermetablic or oxide, the nucleation of that phase can occur prior to cooling below the nominal alloy liquidus. When this occurs in such a fashion that

nucleation occurs without significant growth, as during very rapid quenching or cooling following the last solid formation, then the resulting microstructure should contain fine particles of the refractory compound in a matrix of the remaining alloy constituents. Liquid phase nucleation would be expected to result in a random, or at least non-unique orientation relationship between the particles and the matrix. However, results obtained from various systems show this is not consistently true. For example, several workers (18,19) have reported an orientation relation between rare earth oxides and the matrix in Ti based alloys. In contrast, a random orientation has been observed between Be rich particles in Al alloys (16,17). The principal benefit of powder/particulate production, in which the dispersoids form, is such that a uniform distribution of strengthening particles can be ensured even in large parts. However, the same considerations that effect chemical inhomogeneities in solid-solid nucleation of precipitates discussed above, can cause nonuniform size and number distributions in RSP material. In addition, the fine microstructure characteristic of RSP can drive grain growth with consequent boundary enhanced solution/dissolution reactions for dispersoids (15).

Improving the strengthening due to substructure is not normally associated with RSP. However, the use of RSP for incorporating of a network of fine precipitates or dispersoids can stabilize the dislocation substructure to higher temperatures than would otherwise occur (20) may be worthy of further consideration.

Altering the grain size and shape is a natural extension of RSP processing due to the refinement caused by multiple nucleation. Powder processing combined with low temperature or short time compaction techniques provide the greatest potential for refining the grain size, especially when ultrafine powders are used as starting material. Retaining fine grain sizes following recrystallization is enhanced when inert dispersoids or second phase precipitates are incorporated in the microstructure. Without these particles the clean microstructures which could be produced by RSP would be more susceptible to grain growth during a high temperature densification procedure due to the initially high surface to volume ratio in either powder or chopped particulate material.

Controlling the texture of alloys using RSP is not normally considered in microstructure engineering although it is potentially useful. The preferred solidification direction in planar flow casting could be used in a built-up structure to maximize the desired overall crystallographic orientation and resulting properties. On the opposite extreme, spherical powder can be sintered or dynamically compacted to provide a random texture if greater isotropy is desired. One application where RSP material was used to produce textured material is the RSR nickel based superalloy radial wafer turbine blade program (21,22). However, the orientations of interest were the working and resulting recrystallization textures produced from the HIP compacted powder. It is not clear whether RSP material was a prerequisite for this application.

APPLICATIONS of RSP to ENGINEERING ALLOY SYSTEMS

The nickel base superalloys have probably benefited most significantly from the application of RSP to microstructure control. Two immediate, and different, examples are IN 100 and Rene' 95. The former alloy system was developed originally as a casting alloy. When the composition was atomized and HIP'ed the microstructural refinement and casting segregation elimination allowed the alloy to be hot worked by extrusion or, more significantly, isothermal forging to near net shape (23). Rene 95 was developed using powder atomization and took advantage from the beginning of such features as casting segregation elimination and solid solubility increases allowing alternative strengthening phases (24).

Refractory peritectic forming elements have been used to develop new aluminum based alloys having excellent high temperature properties. The systems Al-Fe-Ce, Al-Fe-Mo and Al-Fe-V-Si have been produced using the powder atomization and planar flow casting techniques and are discussed in this conference. The

microstructural effects of these refractory additions is to form relatively stable intermetallic compound dispersoids during the initial cooling from the melt which do not coarsen significantly during subsequent thermal-mechanical processing. Retention of the uniformly distributed fine particles is crucial to attaining the improved mechanical response at high temperatures.

The use of RSP techniques to produce tool steels with significantly improved properties have been documented (25). The primary microstructural benefit of RSP processing is the refinement of the grain size and the uniform distribution of the precipitate forming elements. The size, spacing, morphology and distribution of carbides and sulfides, has been correlated with the fracture process in a variety of tool steels (26).

In titanium alloys, the addition of rare earth metals or oxides have been successful in incorporating a fine distribution of oxide dispersoids (27). The gettering ability of pure rare earth metals allows the formation of oxide dispersoids in these alloys to produce lower interstitial oxygen titanium alloys with potentially superior ductilities. The coarsening behavior of these dispersoids, especially during grain growth associated with compaction operations, is of major concern since precipitate free zones have been observed in the vicinity of mobile boundaries (15).

CONCLUSIONS

The above discussion permits the following conclusions to be drawn:

- RSP has the potential to enable microstructural engineering of advanced metallic materials. However, there are numerous decisions which must be made in the selection of consolidation processing and alloy composition.
- The major benefits provided by RSP are the capability to produce materials with fine grains and which contain a uniform distribution of dispersoids.
- The behavior of materials with very fine grains (d ≤ 1µm) may not follow the Hall-Petch relationship.with regard to strength. These materials also may not be as tough as somewhat (albeit fine in a conventional sense) coarser grained materials due to the propensity for strain localization.
- In the natural hierarchy of size of microstructural features, RSP can be used to affect those which have dimensions of a few µm or less.
- Many microstructural features affect different properties differently. Thus a priori knowledge of the pacing property or the relative importance of various properties is essential for a given application.
- The benefits realized from dispersoids in ductile materials may not translate into brittle materials such as intermetallic compounds.

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